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EFFECT OF SULFUR, OXYGEN,
AND HYDROGEN SULFIDE SURFACE FILMS
ON THE ADHESION OF CLEAN IRON

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16. Abstract Adhesion experiments were conducted with the (011) crystal face of iron in contact with iron. The surfaces were examined clean and with sulfur, oxygen, and hydrogen sulfide present. Both LEED and Auger analyses were used to determine the nature of surface changes that occurred with the adsorption of gases on that surface. Sulfur and hydrogen sulfide afford greater resistance to adhesion of iron to itself that does oxygen with an equivalent surface coverage. The greater the surface coverage with hydrogen sulfide, the lower the force of adhesion. With hydrogen sulfide, three structures were observed: a(2x4), a (1x2), and a full monolayer that had a close-packed arrangement.			
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EFFECT OF SULFUR, OXYGEN, AND HYDROGEN SULFIDE SURFACE FILMS ON THE ADHESION OF CLEAN IRON

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SUMMARY

An investigation was conducted to determine the influence of a sulfur-containing gas on the adhesion of iron (011) surfaces. Experiments were also done with equivalent surface coverages of sulfur, oxygen, and the sulfur-containing gas hydrogen sulfide to determine which had the greatest influence on adhesion. The studies were made in a vacuum apparatus containing both LEED and an Auger spectrometer to determine the presence and arrangement of sulfur, oxygen, and hydrogen sulfide on the iron surface before and after adhesive contact. A 3.0-millimeter-diameter flat contacted a larger (8.0-mm diam) iron (011) surface under applied forces of from 20 to 350 dynes (2×10^{-4} to 3.5×10^{-3} N). All adhesion experiments were conducted at 10^{-10} to 10^{-11} torr.

The results of the investigation indicated that adsorbed sulfur and hydrogen sulfide exert a stronger influence on the adhesion of one iron surface to another than does oxygen. At a given applied force, increasing surface coverage with hydrogen sulfide reduced the adhesive force. An Auger analysis revealed that sulfur causes greater difficulty in obtaining and maintaining a clean iron surface than does carbon.

INTRODUCTION

In the construction of mechanical components for lubrication systems, the most commonly used materials are iron-base alloys. The chemical interaction of iron with constituents of the environment and the lubricating media plays a very important role in determining the adhesion, friction, and wear behavior of these iron alloy mechanical components. Considerable research effort has been expended in attempts to gain an understanding of the nature of these chemical interactions. The difficulty is that alloys and/or chemically complex lubricating media have been used for these studies. Such multicomponent systems make analysis of basic interactions difficult. The exact role,

for example, of oxygen and sulfur in the adhesion, friction, and wear of iron-base materials is still not completely understood.

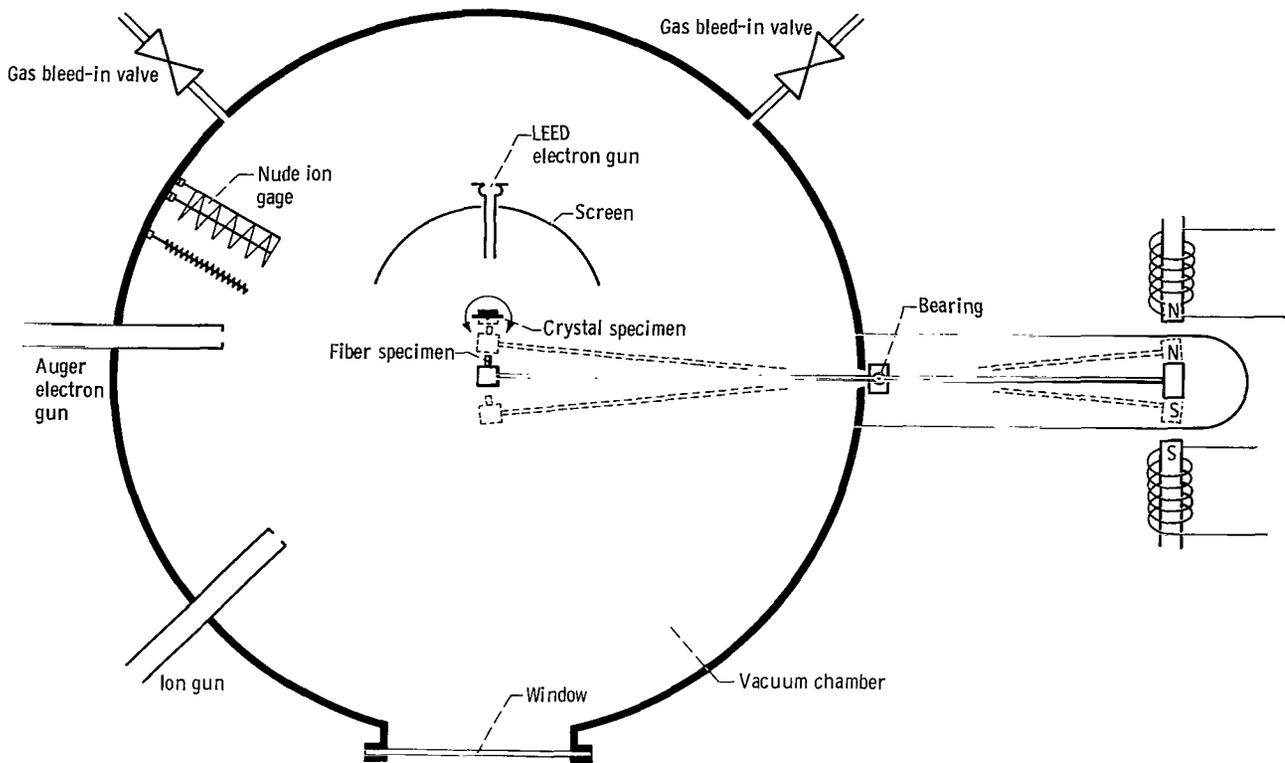
The most advantageous method of studying the interaction of iron with such chemically active species as oxygen and sulfur present in lubrication systems is to start with a two-component system, iron and oxygen or iron and sulfur. The effect of these species on one of the properties used to determine lubricating effectiveness (namely, adhesion) can then be measured.

Although iron-base materials are the most widely used alloys in the lubrication field, and in other areas as well, very little has been done in the field of surface studies with this single metallic element. A limited amount of field ion microscopy (refs. 1 to 4) and very limited LEED (low-energy electron diffraction) studies have been conducted with iron (refs. 5 and 6). One of the reasons researchers have avoided this metal is the difficulty in getting a material pure enough to obtain a clean surface. The presence of minor impurity elements in iron prevents achieving a clean surface, and crystal transformation severely limits the method used for cleaning when single crystal surfaces are to be studied.

The objectives of this investigation were (1) to obtain a clean iron surface, (2) to determine the adhesion behavior of clean iron single crystals in contact, and (3) to study the influence of films of oxygen, sulfur, or hydrogen sulfide on the adhesion of clean iron. The iron surfaces examined in this study were single crystals, each having the (011) orientation. This particular plane was selected because it is the most densely packed plane of iron and, therefore, the most resistant to deformation. Adhesion experiments were conducted with a 3.0-millimeter-diameter flat iron surface contacting a larger 8.0-millimeter-diameter crystal flat. Forces applied to the surfaces in contact varied from 20 to 350 dynes (2×10^{-4} to 3.5×10^{-3} N) over the 3.0-millimeter-diameter apparent contact area. The clean iron surface was subsequently exposed to oxygen or hydrogen sulfide, and adhesive contact was made with various chemisorbed structures of these gases present on the iron surface. Surface structure was determined by LEED, and elements present on the surface were identified by Auger emission spectroscopy.

APPARATUS

The apparatus used in these studies is shown schematically in figure 1. The single crystal surface mounted in the center of the chamber could be rotated 360° . This rotatability allowed for the making of adhesion measurements on the crystal surface shown in figure 1. Further rotation made it possible to obtain both an Auger analysis and a LEED pattern from the crystal surface in the adhesion contact area. The crystal could also be moved in the lateral and vertical directions.



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Figure 1. - Low-energy electron-diffraction (LEED) adhesion apparatus.

The crystal specimen was supported in the chamber by means of two metal rods (insulated) that were used to resistance heat the crystal. A 100-ampere ac power supply was used for resistance heating.

The 3.0-millimeter-diameter flat-ended iron single crystal, which contacted the single crystal metal surface, was mounted in a stainless-steel holder that was, in turn, mounted to a 1.5-millimeter-diameter stainless-steel beam. The beam was mounted in a bearing-containing yoke. At the end of the beam beyond the pivot point and opposite the 3.0-millimeter-diameter specimen was a small permanent magnet. Outside the chamber wall were two electromagnets. The permanent magnet and electromagnets were positioned in such a manner that like poles faced each other. A variation in the current applied to the magnets could be used to move the beam.

The current applied to the electromagnets was calibrated in terms of the force applied in the adhesion experiments. Load applied to the surfaces in contact was measured by current, as was the force required to separate the crystal surfaces.

The basic LEED and Auger systems were obtained commercially. The LEED electron optics and the vacuum system were of the standard type used by those engaged in LEED studies. The Auger spectrometer gun was located at a position 90° from the

LEED gun (see fig. 1). The electron optics of the LEED gun were of the Varian three-grid type with a fourth grid added for Auger analysis. The LEED beam diameter was 0.6 millimeter. The vacuum system consisted of vacsorb pumps, an ion pump, and a sublimation pump. The system pressure was measured with a nude ion gage, and all experiments were conducted with the vacuum system in the pressure range from 10^{-11} to 10^{-10} torr. No cryopumping was used.

EXPERIMENTAL PROCEDURE

The iron crystals used in this study were cut from a single crystal rod into specimens 8.0 millimeters in diameter and 6.0 millimeters thick. The second contacting crystal was 3.0 millimeters in diameter and 6.00 millimeters long. The crystal orientations were checked after electropolishing, and the specimens were placed in a vacuum tube furnace. They were heated to 600° C and held under vacuum for 24 hours, at which time hydrogen gas was admitted. The system was reevacuated. This procedure was repeated for a 2-week period. The purpose of heating was to attempt to drive carbon and sulfur in the bulk to the surface. The admitted hydrogen was to remove the carbon and sulfur from the surface. After this treatment, the crystals were held in vacuum at 800° C for a prolonged period to remove hydrogen from the iron.

When the aforementioned process was completed, the crystals were removed from the vacuum tube furnace, repolished, and electropolished. They were then placed in the apparatus for adhesion, LEED, and Auger studies.

RESULTS AND DISCUSSION

Clean Iron

The iron crystals were ion bombarded in the apparatus of figure 1 with an ion gun using 600 volts and argon at a pressure of 8.0×10^{-5} torr. The crystal was subsequently annealed by heating it to 500° C for 30 minutes. After cooling to 20° C, both an Auger analysis and a LEED pattern were obtained from the crystal surface.

The Auger analysis of the specimen surface is presented in figure 2. The derivative of the secondary electron energy distribution dN/dV is plotted for various electron energies (eV). The elements identified are sulfur, carbon, oxygen, and iron. The presence of carbon and sulfur on the iron surface is not surprising since other investigators have seen these elements on iron (ref. 7), steel (refs. 7 and 8), and stainless steel (refs. 9 and 10). The carbon and oxygen are believed to be present as adsorbed carbon monoxide. After repeated ion bombardments with argon being bled into the

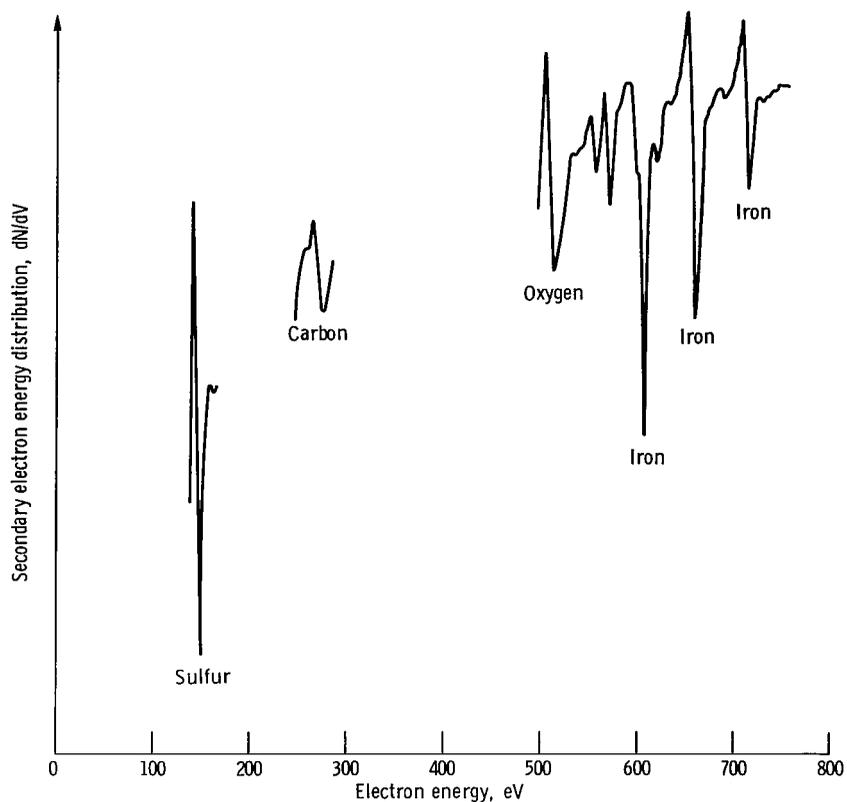
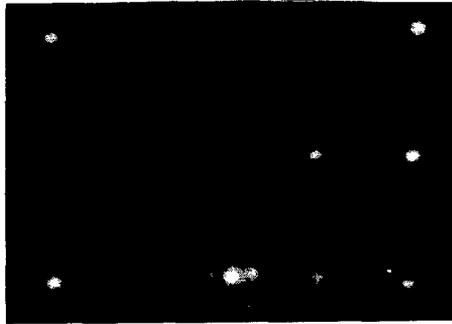


Figure 2. - Auger electron spectrometer analysis of iron (011) surface.

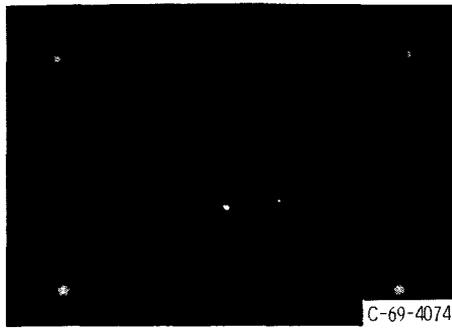
vacuum system to reduce residual carbon monoxide, both the carbon and oxygen could be forced to disappear, leaving only sulfur. A similar observation has been made with 304 stainless steel (ref. 9) and nickel (ref. 11).

The LEED pattern obtained for the (011) iron surface before removal of the carbon and oxygen is shown in figure 3(a). In addition to the diffraction spots associated with the iron (011) surface, additional spots in the pattern indicate the presence of the surface contaminants. Repeated heating, ion bombardment, and subsequent annealing finally resulted in a clean iron surface. The fact that the iron surface was clean was deduced from Auger analysis, the LEED patterns, and LEED intensity maxima.

The Auger analysis of the surface after repeated ion bombardment and annealing indicated only iron on the surface within the sensitivity of the Auger spectrometer. The LEED pattern for the clean iron surface is shown in figure 3(b). The four diffraction spots of figure 3(b) are those that should be observed for clean iron. The atomic model for the (011) surface is presented in figure 4. The unit mesh of figure 4 is reflected in the pattern of figure 3(b). The central diffraction spot does not show because the crystal is between the screen and the window. The absence of any additional diffraction spots further substantiates a clean iron surface.



(a) Iron (011) surface before cleaning. Sulfur and carbon monoxide present on surface.



(b) Clean iron (011) surface.

Figure 3. - LEED patterns obtained before and after cleaning of iron (011) surface.

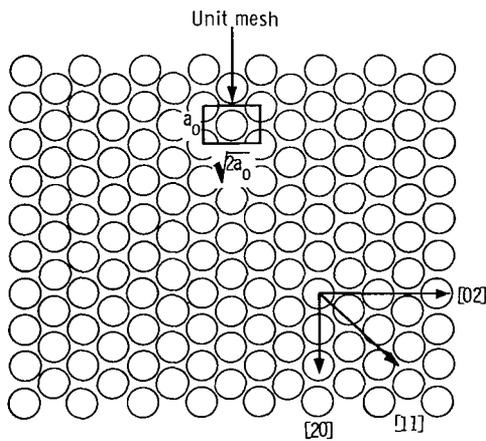


Figure 4. - Atomic model of iron (011) surface.

TABLE I. - INTENSITY MAXIMA FOR
CLEAN IRON (011) SURFACE
(11 REFLECTION)

Kinematic diffraction theory (Laue beams)	Pignocco and Pellissier (ref. 6)	Present study
Beam energy, V		
70	---	58
124	113	113
195	175	185
295	268	265

The characteristic intensity maxima for clean iron from diffraction theory are presented in table I. Also presented are the intensity maxima measured for the iron (011) surface (ref. 6) together with measurements from the present study. The differences between observed and calculated values may result, as suggested in reference 5, from the interaction of the electron beams with electrostatic fields that exist within the crystal surface causing refraction of the diffracted beam. Under such conditions, the wavelength characteristics for the diffracted beam are represented as $[150.4/(V + \psi)]^{1/2}$, where V is the accelerating voltage of the incident beam and ψ is the inner potential of the surface. The values obtained in this study are in fairly good agreement with those of reference 6 and, also, with an allowance of 11 volts for the inner potential, are in good agreement with those from diffraction theory.

Based on Auger analysis, LEED patterns and intensity maxima, the iron (011) surface used in these studies was determined to be atomically clean. Furthermore, Auger analysis indicated that the presence of sulfur may be of equal or greater importance in obtaining a clean iron surface than is carbon.

Adhesion experiments were conducted with the clean iron (011) surface. Simple touch contact of the clean iron was sufficient to give extremely strong adhesive forces. The forces necessary to separate the surfaces exceeded the measuring capabilities of the apparatus (>400 dynes or $>4 \times 10^{-3}$ N). LEED examination of the contact surface region after adhesive contact revealed evidence of strain. The diffraction spots became elongated. Very strong adhesion of clean polycrystalline iron surfaces has been observed in another earlier investigation with polycrystalline iron (ref. 12). These results were, therefore, as anticipated.

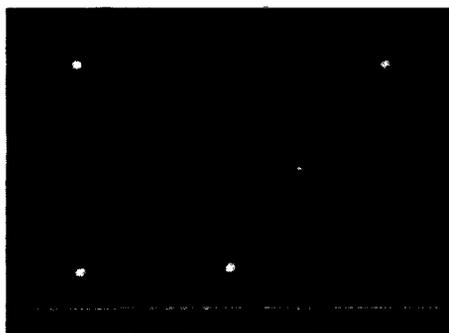
When two clean like pairs of metals are brought into contact, cohesive metallic bonding should occur when the Fermi surfaces are sufficiently close. The energy of

the resulting bonds will be reflected by the cohesive energy of the metal itself. The cohesive bonding force for the clean surfaces will be determined by the cohesive energy of bonding of the material, in this case iron, and the true area in contact.

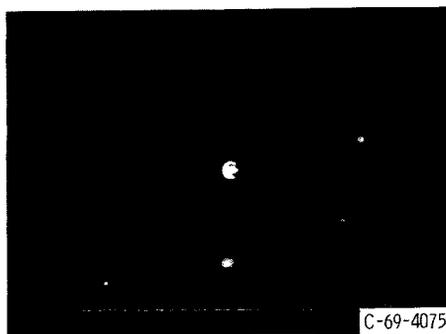
Sulfur at Iron Surface

If the clean iron (Fe) surface is heated to 500^o C and held at that temperature for a sufficient period of time, sufficient sulfur (S) diffuses from within the iron to the surface to produce the Fe(011) c(2×4)-S structure shown in figure 5(a). Auger analysis substantiated that it was sulfur. The possible sulfur sites are indicated in figure 6. The sulfur has two times the lattice spacing of the iron in the [20] direction and four times the lattice spacing in the [02] direction.

The structure Fe(011) c(2×4)-S defines the substrate structure, the arrangement of the sulfur, and the lattice spacing of the sulfur with respect to the iron. The Fe(011)



(a) Diffusion of sulfur to iron (011) surface after heating to 500^o C, producing iron (011) c(2×4)-S structure.



(b) Same surface shown in (a) after adhesive contact under 360-dyne (3.6×10^{-3} -N) force applied over 3.0-millimeter-diameter contacting iron flat.

Figure 5. - LEED patterns obtained with sulfur at iron (011) surface before and after adhesive contact.

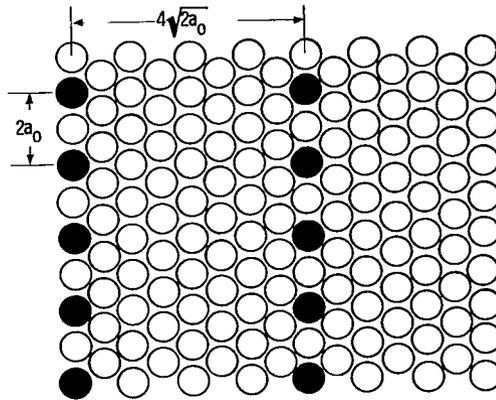


Figure 6. - Sulfur diffused to iron surface, producing Fe (011) c(2x4)-S suggested atomic arrangement.

indicates the high-atomic-density iron atomic plane (011). The atoms of this plane are shown in figure 4. The c in the surface structure designation indicates that the sulfur structure is centered. The (2x4) designation indicates that sulfur has $2a_0$ lattice spacing in the [20] direction and $4\sqrt{2}a_0$ in the [02] direction (see fig. 4 in reference to fig. 6). The S following the (2 x 4) simply indicates that the species is sulfur. The nomenclature used herein to describe surface films is widely used in the literature and was set forth originally in reference 13.

Adhesive contact was made to the Fe(011) c(2x4)-S structure. The first effect observed with the sulfur present on the surface was that it appreciably reduced the adhesive force of iron to itself. The second observation was that the adhesive force

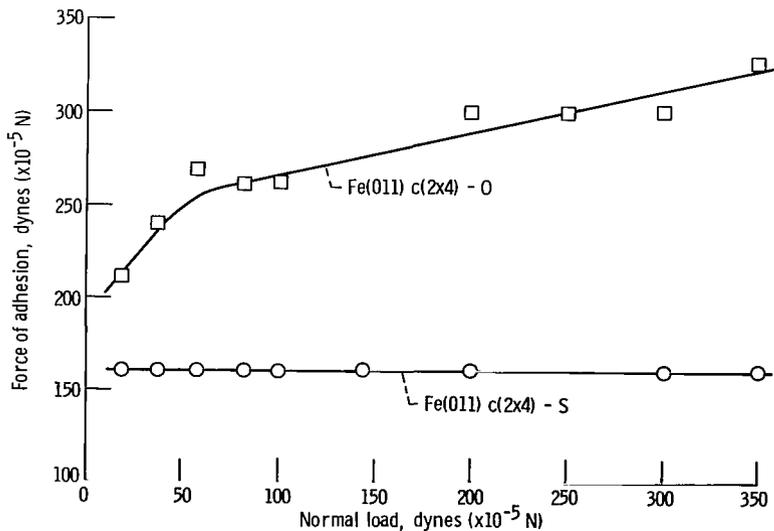


Figure 7. - Effect of oxygen and sulfur on adhesion of iron (011) to itself. Diameter of contacting flat, 3.0 millimeters; contact time, 10 seconds.

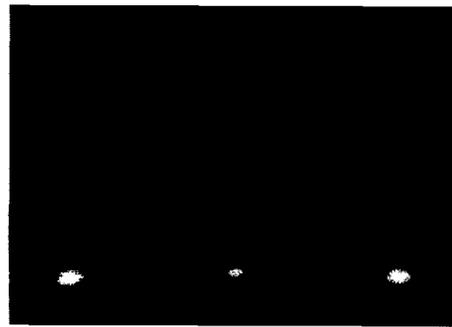
measured was relatively independent of load over a range of loads examined. The data for adhesive forces measured at various applied forces are shown in figure 7 (see curve for Fe(011) c(2×4)-S). A LEED pattern of the surface after adhesive contact is shown in figure 5(b). The surface atomic structural arrangement had undergone a change as a result of the adhesive contact. It would appear that small amounts of bulk contaminants in a metal such as iron can, when diffused to the surface, markedly alter adhesion behavior.

Oxygen on Iron

The clean iron surface was exposed to various amounts of oxygen gas to produce the same surface concentration of oxygen on the iron (011) surface as that with sulfur present (fig. 5). After a number of unsuccessful attempts with too much or too little oxygen on the surface and an exposure of 0.1 Langmuir, a Fe(011) c(2×4)-O structure was obtained and adhesive contact to that surface made. The adhesion results obtained for various applied forces are presented in figure 7. The adhesion forces measured for the oxygen-containing surface were higher at all applied forces than were the values obtained on the sulfur-containing surface. Further, an increase in applied force on contact resulted in an increased force required for separation. This dependency of adhesion force on applied force indicates that an increase in iron cohesive bonding has occurred across the interface with increasing loads. The insensitivity of the sulfur-containing surface to applied loads may indicate that the atomic size of the surface-contaminating atom may exert some influence on adhesive behavior. The sulfur atom is more than twice the size of the oxygen atom and with both having a 2×4 structure on the iron, the amount of exposed iron per unit area available for cohesive bonding with sulfur present on the surface could be expected to be less.

Hydrogen Sulfide Adsorption

Hydrogen sulfide gas was admitted to the vacuum system in sufficient amount to produce a 2×4 structure. The exposure required to obtain the 2×4 structure was 1.0 Langmuir. The exposure in terms of coverage will be influenced very markedly by the position of the gas outlet tube with respect to the crystal surface. When one investigator places the gas outlet tube very close to the crystal surface, a Langmuir value for a specific surface coverage will be markedly different from that obtained when the tube outlet is positioned in the chamber such that the incoming gas does not directly impinge on the crystal surface.



(a) Fe(011) c(2x4)-H₂S.



(b) Surface structure in (a) after adhesive contact with itself under 360-dyne (3.6×10^{-3} -N) force applied to 3.0-millimeter-diameter flat.

Figure 8. - LEED patterns obtained from iron (011) surface after exposure to hydrogen sulfide.

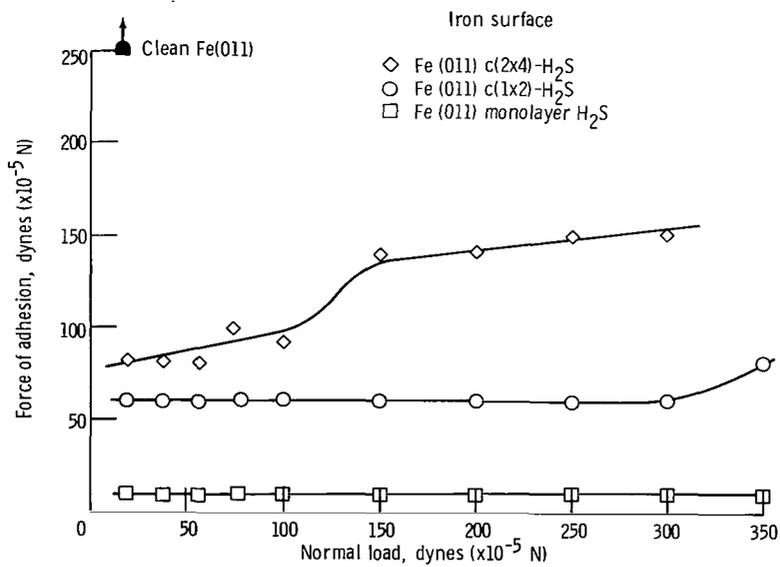


Figure 9. - Influence of hydrogen sulfide adsorption on adhesion of iron (011) surfaces. Diameter of contacting flat, 3.0 millimeters; contact time, 10 seconds.

Sufficient hydrogen sulfide was admitted to the system to produce a Fe(011) $c(2 \times 4)$ -H₂S structure. The resulting LEED pattern obtained is shown in figure 8(a). Adhesive contact was made to the surface, and the LEED pattern after adhesive contact is shown in figure 8(b). A considerable change in background intensity occurred as a result of adhesive contact.

Adhesive forces were measured at various applied forces for the Fe(011) $c(2 \times 4)$ -H₂S structure, and the results obtained are presented in figure 9 (see curve for Fe(011) $c(2 \times 4)$ -H₂S). The force of adhesion increased with increasing load. This result should be compared with that obtained for sulfur in the surficial layer and oxygen on the surface in figure 7. It appears that, for equivalent surface coverages, namely, a (2×4) structure on the (011) surface of iron, chemisorbed hydrogen sulfide affords the greatest surface resistance to adhesion. Sulfur offers intermediate and oxygen the least surface protection.

The lower adhesive forces of iron bonding to iron in the presence of hydrogen sulfide may in part be a steric effect. An examination of atomic attractive energy between like atoms reveals that it is a function of interatomic spacing, as shown in figure 10. In figure 10, *a* is the equilibrium position where the stable cohesive bond is formed. As

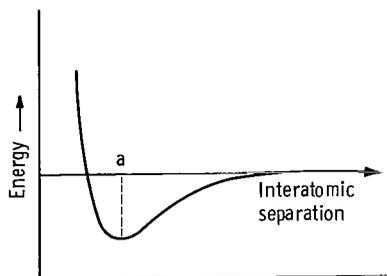
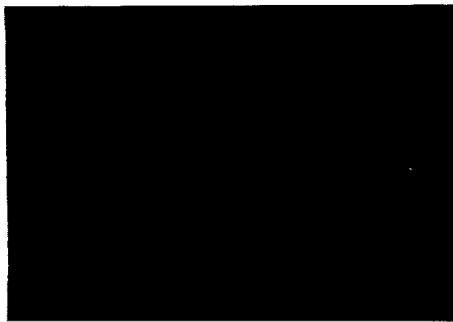


Figure 10. - Attractive energy of cohesive bonding.

the interatomic separation increases, the cohesive bond strength decreases. This simple steric concept is, however, complicated by the fact that chemical bonding of unoccupied iron sites on one of the two reacting surfaces may occur with oxygen, sulfur, or hydrogen sulfide present on the other surfaces.

Greater exposures of the iron (011) surface to hydrogen sulfide resulted in the development of an Fe(011) $c(1 \times 2)$ -H₂S structure, as shown in figure 11. The development of the 1×2 structure required a hydrogen sulfide exposure of 10 Langmuirs. The suggested arrangement of the hydrogen sulfide on the iron (011) surface is shown in figure 12. Adhesion measurements were made for this surface at various loads, and the results obtained are presented in figure 9 (see curve for Fe(011) $c(1 \times 2)$ -H₂S). With greater surface coverage, the adhesive force decreased from the values obtained with



(a) Fe(011) c(1x2)-H₂S.



(b) Iron (011) surface with full monolayer coverage of H₂S.

Figure 11. - LEED patterns obtained with exposure of iron (011) surface to hydrogen sulfide.

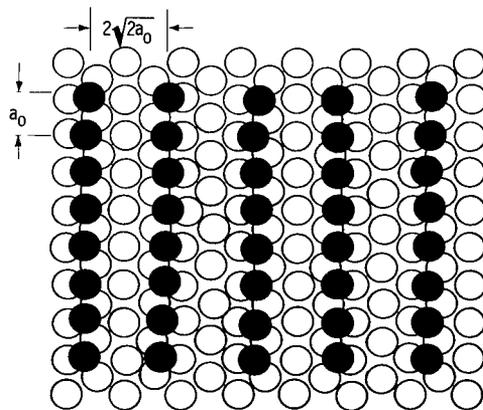


Figure 12. - Suggested arrangement of hydrogen sulfide in (1x2) structure on iron (011) surface.

the (2×4) surface coverage, as might be anticipated. Further, there appears to be a greater independence of contact force applied.

With prolonged exposures of the iron surface to hydrogen sulfide (50 Langmuirs), full monolayer coverage of the iron (011) surface was observed. The surface structure produced a close-packed surface arrangement, as shown by the LEED photograph of figure 10(b). The suggested arrangement of the hydrogen sulfide on the iron (011) surface is shown in figure 13. Further exposures to as much as 100 Langmuirs produced

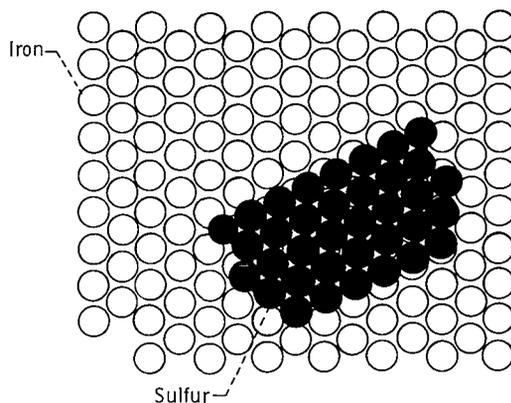


Figure 13. - Suggested arrangement of hydrogen sulfide on iron (011) surface with monolayer coverage. Sulfur coverage is over entire surface although only small portion is shown.

no change in the surface structure. The adhesion forces to this surface were the least of those measured, as shown by the data of figure 9. These data indicate that adhesive force is a function of surface coverage - the greater the coverage, the lower the adhesive force.

Subsequent heating of the Fe(011)-(1×1)-H₂S surface to 500° C did not produce a change in the surface arrangement but simply intensified the diffraction spots. Heating to this temperature should produce a decomposition of the hydrogen sulfide to form iron sulfide and liberate hydrogen. The pressure in the system rose, and diffraction peak intensities changed, indicating the liberation of hydrogen. Adhesion measurements revealed no change in the adhesion forces. These results suggest that the adsorbed close-packed monolayer of hydrogen sulfide is present on the iron (011) surface with the sulfur directly in contact with the iron and the hydrogen in an upper layer. Decomposition simply removes the hydrogen layer, leaving the sulfur close-packed layer on the iron surface.

SUMMARY OF RESULTS

Based on the data from adhesion studies and the analysis of an iron (011) surface with LEED and Auger in the presence and absence of sulfur, oxygen, and hydrogen sulfide films, the following results were obtained:

1. Sulfur and hydrogen sulfide present on iron in a (2×4) structure exert a greater influence than oxygen on the reduction of the adhesion of iron to itself, with equivalent surface coverage.

2. The adhesion force decreased with increased surface coverage of hydrogen sulfide on the iron surface. Monolayer coverage of the iron (011) surface afforded the greatest reduction in adhesion.

3. With hydrogen sulfide adsorption, three surface structures were observed, a (2×4), a (1×2), and a full monolayer coverage that formed a close-packed layer on the surface.

4. Auger analysis of an iron surface in the process of cleaning revealed that the principal material that diffuses to the surface to make obtaining a clean surface difficult is sulfur, rather than carbon as previously believed.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, December 9, 1969,
129-03.

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